OXFORD CRYOSYSTEMS

Cryostream 700 Series, Plus and Compact

Operation & Instruction Guide

OXFORD CRYOSYSTEMS CRYOSTREAM

Operation & Instruction Guide v6.3

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1 Introduction

Welcome to the Oxford Cryosystems Cryostream Operating and Instruction guide. This guide is relevant for the 700 Series Cryostream, Cryostream Plus and Cryostream Compact.

The Cryostream is the world's leading nitrogen gas low temperature attachment for x-ray crystallography. It can be used for all applications from shock cooling to lengthy data sets lasting many weeks. Its versatility and flexibility means it can be fitted to practically any x-ray system including an Image Plate System, CCD Detector, Eulerian Cradle Four Circle Diffractometer, Kappa Diffractometer or Powder Diffractometer. Due to its unique design, the Cryostream operates from 80 K to 400 K (or 80 to 500 K for the Compact and Plus versions), with a stability of 0.1 K and low liquid nitrogen consumption of 0.6 L per hour at 5 L/min of gas flow.

Before using the Cryostream, please read the section, 'Liquid and Gaseous Nitrogen Safety Sheet'.

1.1 Please register your Cryostream!

In order to help us provide technical support, we need you to register your Cryostream system. You will need to fill in the **WARRANTY CERTIFICATE** attached at the end of this manual and faxing or emailing this back to us. This is very important as it allows us to track your enquiry and tie this up with the technical notes we have on your particular system.

1.2 How the Cryostream works

Figure 1 below illustrates the liquid and gaseous nitrogen flow circuit of the Cryostream.

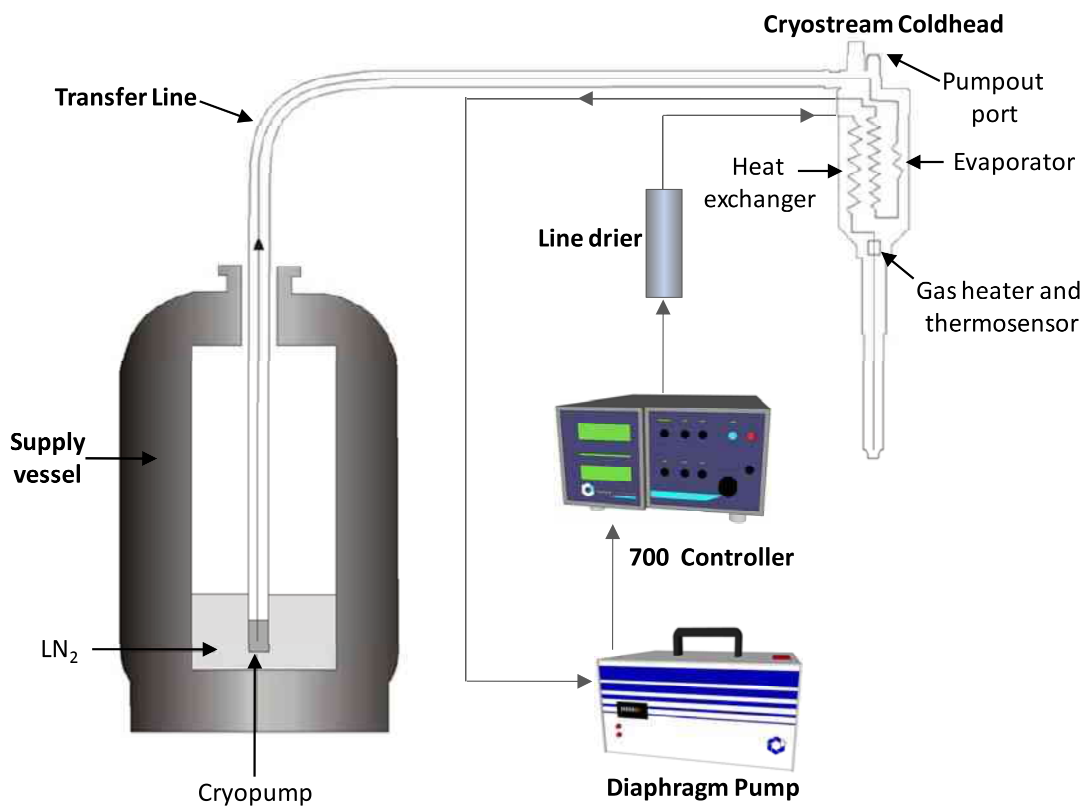


Figure 1 – Layout of a Cryostream

Liquid nitrogen is drawn up by the action of the diaphragm pump from an unpressurised supply vessel, through a flexible vacuum insulated transfer line, into the Cryostream Coldhead. The supply vessel can be any convenient container of liquid nitrogen such as a conventional metal Dewar. Please note that the Dewar vessel is *not* sealed.

The liquid nitrogen, once inside the Cryostream Coldhead, passes through a heater called the evaporator heater, which evaporates most of the liquid into vapour at the boiling point of liquid nitrogen. This vapour then flows outward along one path of the heat exchanger, through the Cryostream temperature Controller, to arrive at the inlet of the diaphragm pump at approximately 10 K below room temperature. The nitrogen gas from the pump may then be dried with a line drier unit if appropriate. The flow rate of the gas from the pump is then regulated by a variable flow Controller in the Cryostream temperature Controller. This gas flows back into the Cryostream Coldhead where it is re-cooled along the second path of the heat exchanger.

Therefore, the main heat exchanger carries the gas streams to and from the constant-flow pump that operates at room temperature. The nitrogen flow rate is set to either 5 or 10 L/min by the variable flow Controller. After returning to the cold end of the heat exchanger, the gas temperature is regulated by a heater and sensor before entering the nozzle of the Cryostream. The gas then flows along the isothermal nozzle and out over the sample. The temperature indicated on the Controller is a mapped temperature for the crystal position (see Section 6.1).

The flexible liquid nitrogen transfer line, the heat exchanger, evaporator heater and delivery nozzle share a common high-vacuum insulation jacket which is pumped out and sealed before use. An adsorption cryopump is built into the end of the rigid part (leg) of the transfer line to increase the strength of the vacuum when plunged into liquid nitrogen.

Note that the Cryostream 700 series is supplied with the 700 series Controller which is designed specifically to manage Oxford Cryosystems range of coolers.

The Cryostream flow rate is variable and can be set to either 5 or 10 L/min of gas, this equates to approximately 0.6 and 1.2 L/hour of liquid nitrogen. This means that a 60 L Dewar will last for more than three days at 5 L/min. It is worth noting that different Dewars have different rates of boil off, so it is worthwhile taking this into consideration when running the Cryostream and leaving it unattended for long periods. Also note that below 90 K, the lower flow rate of 5 L/min is slowly increased to achieve the very low temperatures.

Because the Cryostream applies the same gas pressure at the crystal as it does in the Dewar, it is safe to replenish the supply of liquid nitrogen to your Dewar at any time without any fluctuations in gas temperature. This can be simply done by pouring or using a mildly pressured secondary storage vessel or an automatic refilling system (available from Oxford Cryosystems).

1.3 Items required for assembling your Cryostream system

The component parts of the system are:

- ✓ The Cryostream Coldhead (700 Series, Plus or Compact) and flexible transfer line (integral)
- ✓ 700 Series Controller
- ✓ A diaphragm gas pump
- ✓ Interconnecting Teflon tube set*, including:
    - Two long tubes (3 m) with fittings
    - Two medium tubes (2 m) with fittings
    - One short piece of tube (0.7 m) with fittings
    - One red dry air tube (3 m) with right angled connector
- ✓ One Coldhead cable
- ✓ One serial cable for Cryopad
- ✓ One Cryopad CD

** Note that the tube lengths may be different for the Cryostream Compact, depending upon specific system configurations.*

Optional extras include:

- ✓ A Varibeam Coldhead support stand
- ✓ An AD51 dry air unit (or alternative dry gas supply)
- ✓ Line drier units
- ✓ Dewar vessel – either an Oxford Cryosystems 60 L Dewar* or a suitable alternative
- ✓ Nozzle alignment tool

** Note that a smaller 13 L or 25 L vessel may be supplied with the Cryostream Compact, depending upon specific system configurations.*

1.4 Items supplied for the Cryostream maintenance

The component maintenance parts supplied are:

- ✓ A CRH25 reactivation heater
- ✓ A pump-out adaptor

2 Setting up

2.1 Operating voltage

It is essential that the Cryostream is configured to operate on the local mains electrical supply. Check the following:

Item	Operating voltage requirements
Cryostream Controller	Switch-mode power supply automatically selects the correct voltage and frequency: 200-240 Volts AC, 50 Hz, 3 Amps 100-120 Volts AC, 50-60 Hz, 6 Amps. Make sure an 'Anti-Surge' (T) type fuse of the correct rating is fitted. The correct fuse is fitted as standard.
Gas Pump Unit	Three voltage variants are currently available: 220-240 Volts AC, 50 Hz 115 Volts AC, 60 Hz 100 Volts AC, 50-60 Hz Make sure you have the correct pump.
CRH25 Reactivation Heater	Two voltage variants are available: 230 Volts AC 115 Volts AC

2.2 Using the Varibeam support stand

The Varibeam is an extremely robust and rigid stand that will support the Cryostream on almost all x-ray systems. The Varibeam has a leadscrew positioner and an X-Y Positioner that allows the Cryostream nozzle to be positioned very accurately at the crystal.

The stand can be assembled in various configurations; the rotation of the horizontal arm; angling the nozzle; and the block gripping the Cryostream can be removed and fitted onto the other side of its support plate. This stand will support and guide the nozzle in all configurations.

2.2.1 Assembly and positioning of the Varibeam Coldhead support stand

The Varibeam column and cross-arm are anodised aluminium to give an extremely tough finish. When the stand is being put together, try to avoid bolting the Varibeam in a position which causes obstruction where access to the crystal is important, collision with any microscopes or circles of the diffractometer, or obstruction to x-ray tube housing.

If you are unsure of exactly where you want to position the Cryostream on your system, try setting it up on a desktop first to give you a chance to consider all the options. Alternatively, use the blue Mounting Pin supplied with the support stand as a guide for aligning the real nozzle. For determining the exact crystal position, simply place a fibre in the small hole at the end of the blue Mounting Pin, or use the Nozzle Alignment Tool.

The Varibeam is supplied with bolts to allow the user to securely fix the stand to the cabinet tabletop. Users are often reluctant to drill holes without first experimenting with various positions first. If this is so, use a G-clamp (C-clamp) to temporarily fix the Varibeam to the tabletop. If the positioning point of the Varibeam base is not close enough to the edge of the table top, try bolting the stand to a flat sheet of metal which can be clamped to the edge of a cabinet top.

In many cases bolting the Varibeam to a cabinet top can prove quite a lot of work or is just not practical, so try using tapping screws to fix the support stand in place. **Make sure the cabinet top is sufficiently strong enough to prevent the tapped screws from tearing from it.**

2.3 Mounting of the Cryostream on the x-ray system

The mounting of the Cryostream depends on the particular x-ray system being used. Oxford Cryosystems recommend the use of the Varibeam support stand to mount the Cryostream on all x-ray systems.

NOTE

The following rules and instructions are guidelines only and if a user has an alternative technique for mounting the Cobra then they are welcome to use it. If the user is in anyway unsure of the mounting of the Cobra they should contact their local Cobra supplier or Oxford Cryosystems for advice.

2.3.1 General rules

There are a few general rules the user should consider when mounting the Cryostream and fixing the support stand in position.

1. Do not point the nitrogen cold stream at the detector.
2. Try to limit the amount the Cryostream nozzle infringes the path of the x-rays.
3. Do not point the cold stream at any optical device or gearing (these devices need to be more than 15 cm away).
4. Do not mount the cold stream coaxial with the goniometer head, this will cause icing problems.
5. Do not fix the stand to your cabinet top so it prevents access to your x-ray tube, prevents the detector being swung in theta or makes access to the crystal difficult.
6. The crystal should be less than 8 mm from the end of the nozzle in the centre 3-4 mm of the gas stream; this will help to prevent icing. The Oxford Cryosystems Nozzle Alignment Tool is ideal for determining the correct position.
7. The transfer line is made out of flexible tubing containing a very fine continuous metal capillary. This should not be flexed to a radius of less than 200 mm so **DO NOT BEND THE FLEXIBLE TRANSFER LINE TOO SHARPLY**. Obviously, the more the transfer line is flexed the more likely it is to fatigue.

2.3.2 Setting up on Eulerian Cradle Four Circle Geometry (e.g. Bruker AXS P4, Stoe Stadi-4)

The Cryostream should be mounted so that the nozzle enters the chi circle over or close to the collimator at an angle of 45º. There may be a need to reduce the movement of the omega circle so that the goniometer does not hit the nozzle of Cryostream. The Varibeam stand should be mounted behind the x-ray housing and toward the back of the cabinet, with respect to the path of the x-rays.

In the Bruker AXS cabinets, there is often a slot in the back for the cooling hose of their CDD Detector. This also makes for a very useful access point for the transfer line. It is likely the Dewar vessel will need to be raised off the floor so the transfer line can reach the bottom of the vessel. This slot is also a very good port for the tubing and Coldhead cable.

2.3.3 Setting up on Kappa Four Circle Geometry

The Varibeam support stand should be mounted either to the left or right of the x-ray housing to reduce interference when accessing the crystal and the movement of the detector. The Cryostream should then be mounted at about 65º. This set up will require a hole to be machined in the cabinet and it is likely the Dewar vessel will need to be raised off the floor so the transfer line can reach the bottom.

2.3.4 Setting up on the Marresearch Image Plate Detector

There is plenty of access into the crystal on the Marresearch image plate system and although there is a temptation to mount the Cryostream vertically over the horizontal goniometer one must be careful to avoid pointing the cold stream at the CCD camera pointing up from underneath. We recommend the user tilts the Cryostream from 90º vertically to about 55º towards the goniometer housing. The position will depend on which side the user wishes to gain access to the crystal. It is also possible to invert the head in order to point the stream upward.

On the Marresearch image plate the crystal is mounted very close to the collimator and often results in the collimator infringing on the dry air shroud or cold stream of the Cryostream. This can cause icing and temperature fluctuations, so it may be necessary to modify the collimator. Do not rush into this modification.

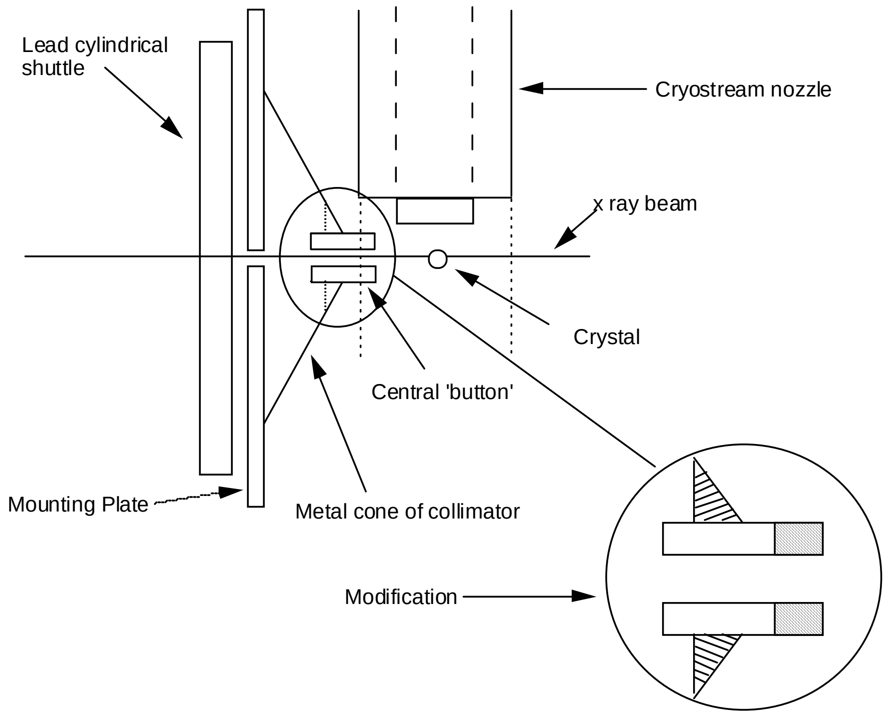


Figure 2 – Modification to a Marresearch Image Plate Collimator

2.3.5 Setting up on the Rigaku R-Axis Image Plate Detector

The Cryostream should be mounted opposite the microscope, perpendicular to the x-ray beam path, at a fairly unrestricted angle of between 55º and horizontal (ideally about 45º). If the R-Axis is fitted with both a CCD camera and a microscope, then one may need to be (re)moved depending on the amount of space available. If the Cryostream is any steeper than 55º the goniometer may interfere with the laminar flow of the cold stream and cause icing to occur. The Varibeam support stand is ideal for this application.

If the R-Axis is fitted with an inverted Phi-Axis, the Cryostream can then be pointed upward at the sample to maintain the pin mount - cold stream orientation.

2.3.6 Setting up on the Bruker SMART / APEX CCD System

The Cryostream should be mounted so the nozzle is as close to vertical as possible. If 90° is vertical, then mount the Coldhead at about 80°. This steep angle is necessary as manufacturers continue to increase the diameter of the detector. The Varibeam stand should be mounted behind the x-ray housing and toward the back of the cabinet, with respect the path of the x-rays.

In the newer Bruker AXS cabinets, there is often a slot in the back for the cooling hose of their CCD Detector. This also makes for a very useful access point for the transfer line. It is likely the Dewar vessel will need to be raised off the floor so the transfer line can reach the bottom of the vessel. This slot is also a very good port for the tubing and Coldhead cable.

2.4 Connecting up the Cryostream

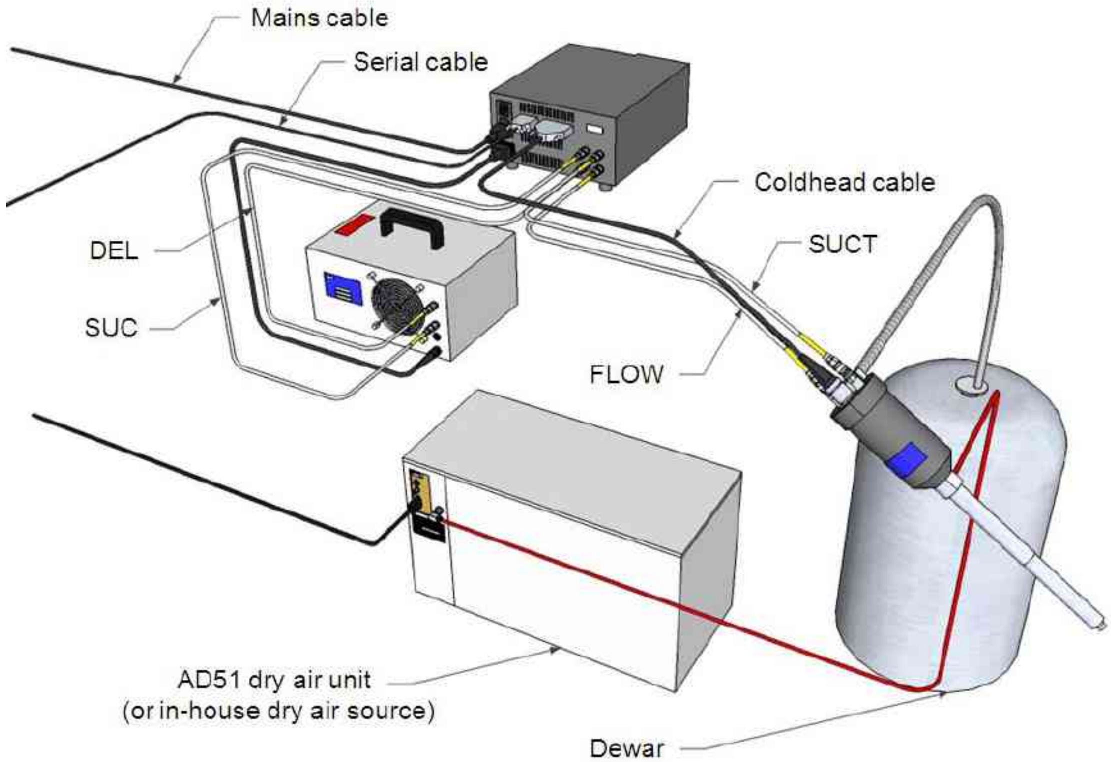


Figure 3 – Tube and cable connection scheme

The diagram above illustrates all the connections that have to be made. The Cryostream consists of six 6 mm (outside diameter) tubes:

1. Two 3 m long tubes
2. Two 2 m long tubes
3. One 0.7 m tube, marked with **GREEN** collars
4. One red tube (3 m) with right angled connector.

2.4.1 The Teflon tubing and connecting the system together

All new Cryostreams are supplied with Quick-Release fittings. In an attempt to make the Cryostream simple to assemble, all the connectors have been marked at each end to match the corresponding fittings on the system. Always ensure the collar clicks into place when connecting the fitting.

IMPORTANT NOTE

Before connecting the system together exactly as illustrated in the diagram and with the information in the table below, the system should be run without a line drier fitted into the circuit.

Table 1 below outlines tube connections:

Tube	Tube marking	Fits onto:
1 (a)	3 m tube with *Coldhead SUCT* label	SUCT Quick Release fitting on the Coldhead
1 (a)	3 m tube with *Controller SUCT* label	SUCT Quick Release fitting on the Controller
1 (b)	3 m tube with *Coldhead FLOW* label	FLOW Quick Release fitting on the Coldhead
1 (b)	3 m tube with *Controller FLOW* label	FLOW Quick Release fitting on the Controller
2 (a)	2 m tube with *Controller SUC* label	SUC Quick Release fitting on the Controller
2 (a)	2 m tube with *Pump SUC* label	SUC Quick Release fitting on the Gas Pump Unit
2 (b)	2 m tube with *Pump DEL* label	DEL Quick Release fitting on the Gas Pump Unit
2 (b)	2 m with *Controller DEL* label	DEL Quick Release fitting on the Controller

Table 1 – Cryostream tube connections

When fitting a line drier unit, firstly unplug the FLOW Quick-Release fitting on the Controller:

2 (b)	2 m tube with *Pump DEL* label	DEL Quick Release fitting on the Gas Pump Unit
2 (b)	2 m with *Controller DEL* label	IN Quick Release fitting on the Line Drier
3	0.7 m tube with Quick-Release fitting end	OUT Quick Release fitting on the Line Drier
3	0.7 m tube with Quick-Release fitting end	DEL Quick Release fitting on the Controller

Table 2 – Cryostream line drier fitting

2.4.2 Connecting up a dry air shroud gas

The system is now set up to deliver nitrogen gas but in order to operate in an ice free environment at low temperatures, the nitrogen gas stream requires a shroud gas as it exits the nozzle. This gas can be either dry air with a dew point of less than -60ºC or an inert gas such as nitrogen that is often fed in and piped around laboratories. Oxford Cryosystems manufacture the AD51 dry air unit, a stand-alone unit that provides a constant stream of clean dry air and is ideal for this application.

Attach the red nylon tube from the tube set to the side connector on the delivery nozzle of the Cryostream, a right angle connector is provided.

Insert the rigid leg at the end of the flexible transfer line into an open Dewar vessel (see next section on Dewar vessels) containing liquid nitrogen. **Always make sure that there is enough nitrogen in the Dewar vessel to cover the bottom 15 cm of the rigid transfer line. This is vital, as the Cryopump in the leg needs to be submerged in liquid nitrogen to ensure a good vacuum.**

Press some insulating material into the neck of the Dewar (a white Teflon bung is provided with the Oxford Cryosystems 60 L Dewar vessel) and around the transfer line in order to minimise boil off and to prevent the development of ice. **However, the Dewar must not be sealed!** More elaborate neck fittings can be constructed, but always remember to vent the Dewar.

2.5 Dewar vessels

The purpose of the Dewar vessel is to hold the liquid nitrogen supply used by the Cryostream. There are a number of variables to consider when deciding on which Dewar vessel to purchase for use with the Cryostream.

- Capacity – this is the most important consideration when deciding on a Dewar. The recommended capacity is between 30 and 60 litres. The liquid nitrogen consumption of the Cryostream does vary with flow rate so make sure the Dewar is topped up.

NOTE

It is important to remember that the rigid transfer line of the Cryostream is plunged into the Dewar and is 900 mm long, so it can only utilise the top 850 mm of a Dewar vessel. It is possible to use a 100 L Dewar, but if the rigid transfer line does not reach the bottom, the Cryostream will not utilise its full capacity.

- Construction – either stainless steel or aluminium.
- Neck opening size – Dewar necks vary in size. If the opening is too small, there may be problems refilling the Dewar. If the Dewar opening is too large, then the rate of boil off will be very high and contaminants will get into the liquid nitrogen.

2.6 Stainless steel line drier units

2.6.1 Before fitting and using your line drier unit

When first installing your Cryostream, **run the system without fitting the line drier**. If there are any inward leaks or a high level of ice in the liquid nitrogen, the Cryostream will block in a few days but will not contaminate the line drier with water. If this occurs, re-check all the Quick-Release fittings, check the liquid nitrogen in the Dewar vessel for ice contamination then rerun the system. The Cryostream should run for about 5-7 days before a block occurs, although poor quality nitrogen can reduce this to 2-3 days (the ALARM Lamp will flash and on pressing the ALARM button the STATUS screen with indicate a pressure warning on the Cryostream). If none of these appear then the quality of the liquid nitrogen is very good.

Once the system has run for about a week, fit your line drier.

2.6.2 Fitting and using your line drier unit

The line drier is designed to remove **traces of water vapour** from the nitrogen stream. **It will not cope with gross contamination** of the nitrogen supply or a large inward leak.

It is recommended to warm up and clean out the liquid nitrogen vessel at regular intervals, and keep the vessel neck covered to prevent atmospheric moisture and other contaminants getting into the Dewar. Also ensure the liquid nitrogen is of good quality.

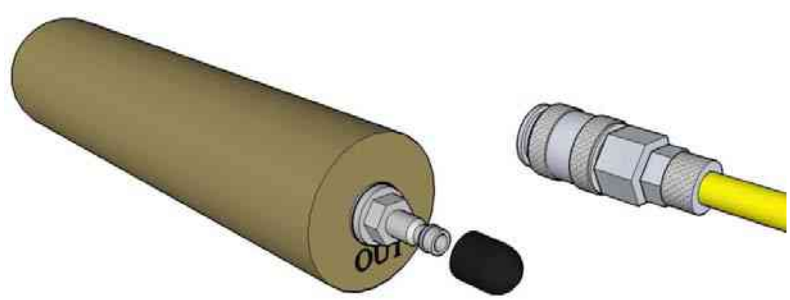


Figure 4 – Line drier unit with blanking cap and connection cable

Remove the blanking caps (these caps must be fitted to the line drier during transit and storage). Fit the line drier unit in the FLOW gas circuit pipe work. This is between the FLOW connector on the

Cryostream Controller and the FLOW connector on the Cryostream Coldhead. **Do not leave the line drier open to the atmosphere as this will cause contamination.**

Eventually the line drier will become saturated with water and require replacing. Saturation of a line drier in the gas circuit may be indicated by a pressure warning in the Controller (see page 41). New line driers are available from Oxford Cryosystems or your local agent.

3 Running the Cryostream

3.1 700 Series Controller

The 700 Series Controller (Figure 5 and Figure 6) is designed to provide a completely flexible means of controlling the Cryostream and is used for the standard 700 series, Plus and Compact versions of the Cryostream.

Figure 5 – 700 Series Controller – front view

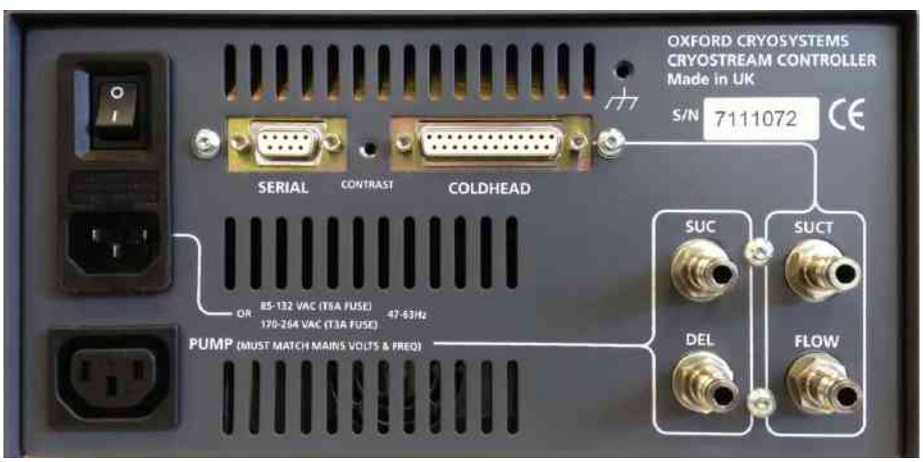


Figure 6 – 700 Series Controller – back view

3.2 How to Switch the Controller On

3.2.1 Final checks before switch on

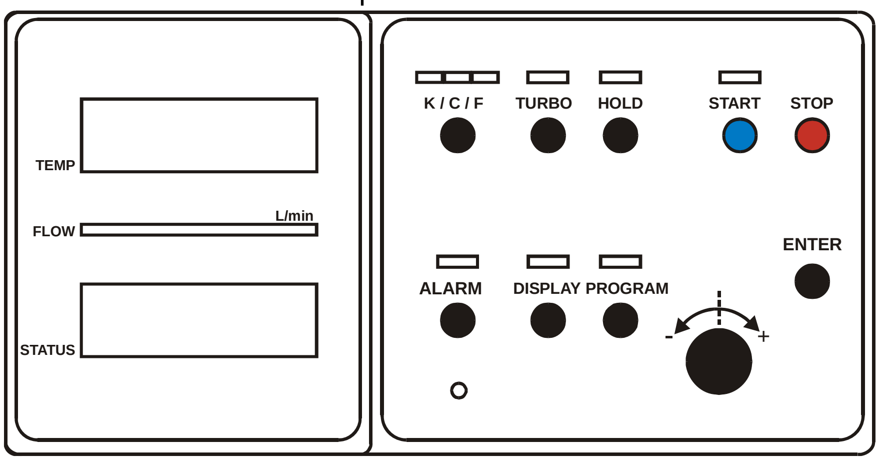
1. All the Teflon tubes are pushed firmly into their Quick-Release Connectors. Each one should be firmly clicked into place.
2. There is sufficient liquid nitrogen in the Dewar and the Cryostream rigid transfer line is in the Dewar vessel.
3. If a dry air shroud is required, it is connected to the Quick-Release connector at the top of the Cryostream nozzle.

Once the system has been connected up correctly and the transfer line has been placed into the liquid nitrogen Dewar vessel, press the 'ON' switch for the Cryostream Controller. This is on the top, left-hand side of the rear panel of the Controller, (right-hand side when viewing from the front of the Controller).

3.3 Cryostream initialisation and screen options

As the Controller is switched on, it undergoes an initialisation process and a self-check procedure. During the self-check, the Cryostream checks to make sure all parts of the Cryostream system are working properly. If a part of the system passes the check, a tick will appear next to it. If the part fails, a cross will appear next to it and a diagnostic message will appear.

3.4 700 Series Controller front panel



The 700 Series Controller front panel includes the following items:

- TEMP screen. Whilst the Cryostream is running this screen displays the temperature of the nitrogen gas stream. During start-up and shutdown the screen is used for status messages.

- FLOW meter. The flow meter indicates the nitrogen gas flow in L/min. If no lamps are illuminated, the gas flow is zero, or less than 3 L/min. Three lamps indicates 5 L/min, all eight lamps indicates 10 L/min.

- STATUS screen. This screen displays information described in detail in Section 3.4.1.

- K/°C/°F button and lamps. The button allows the temperature units used by Cryostream to be switched between Kelvin, Centigrade and Fahrenheit **at any time**. The current choice of unit is indicated by the illuminated K/°C/°F lamp. For the purposes of this manual, temperatures are indicated in Kelvin (K).

- TURBO button and lamp. The button allows the nitrogen gas flow rate to be adjusted between a normal value (5 L/min except at temperatures below 90 K) and a 'Turbo' value (10 L/min except above 310 K). If the TURBO lamp is OFF this indicates normal flow, whereas ON indicates Turbo flow. In addition, during a COOL phase the Turbo lamp will flash. This indicates a 'Machine Turbo', in which the Cryostream fixes the flow rate at the Turbo value in order to minimise the cooling time.

- HOLD button and lamp. Pressing the HOLD button will execute a Hold (see Section 4) and illuminate the HOLD lamp. If Cryostream is already in a Hold, pressing HOLD again will release it.

- START button and lamp. The START button switches the Cryostream on, executing the start-up phase or the current Phase Table (See Section 4). This button is also used to re-start the control program after it has been halted.

- STOP button. The STOP button will immediately halt the Cryostream, turning off the pump and all the heaters. The Controller may then be safely switched off, or else re-started by pressing START.

> **NOTE**
>
> The approved method of shut down is via an END phase

- ALARM button and lamp. If an alarm condition develops (see Section 4.2.4), the ALARM will be illuminated and a buzzer may sound. Pressing the ALARM button will display the cause of the alarm in the STATUS screen and will also cancel the buzzer.

- DISPLAY button and lamp. The DISPLAY button is used to toggle the Display Mode (see Section 3.4.1), indicated by the corresponding lamp.

- PROGRAM button and lamp. The PROGRAM button is used to toggle Program Mode (see Section 4), indicated by the corresponding lamp.

- IntelliKnob. This knob is used to scroll the contents of the STATUS screen. In Program Mode it is also used together with the ENTER button to input information. The IntelliKnob is speed sensitive. This means the faster the knob is turned, the greater the increment in the number and the slower the knob is turned, the smaller the increment in the number.

- ENTER button. This button is used during Program Mode (see Section 4) to input information.

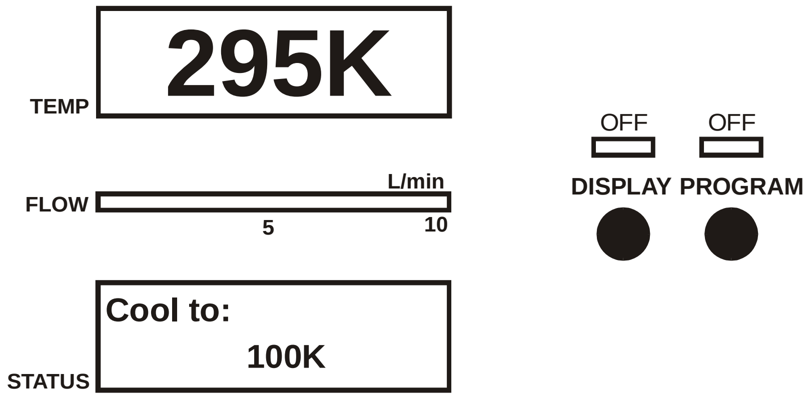
3.4.1 Display Modes

The STATUS screen displays a variety of information depending on the Display Mode and on whether Cryostream is **Running** or **Idle**. In each case the contents of the STATUS screen may be scrolled using the IntelliKnob.

The various situations are summarised below.

3.4.1.1 DISPLAY MODE 1

Cryostream State: **IDLE (Power on, not running)**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **OFF**
Description:



Idle phase table mode

If a program has not been entered, use the IntelliKnob to adjust the temperature and press START to begin. The Cryostream will then enter a COOL function and achieve the required temperature as quickly as possible. This is done by using **Machine Turbo** where the system **automatically** increases the flow to 10 L/min and cools the system down.

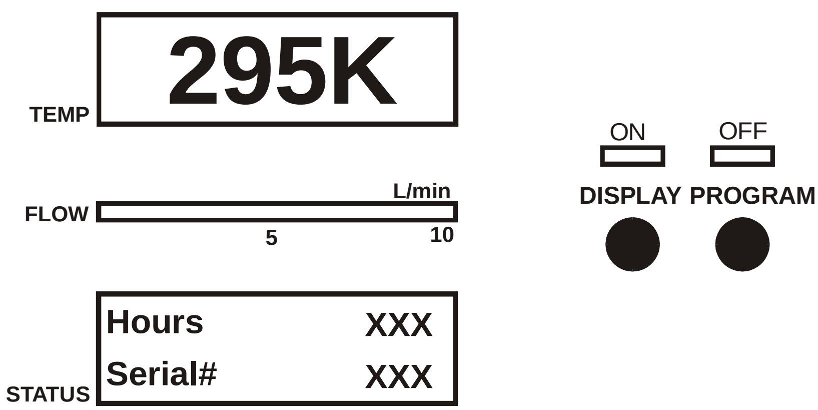
If a program has been entered, use the IntelliKnob to scroll the STATUS screen and press START to begin.

Press DISPLAY or PROGRAM to enter the modes below.

3.4.1.2 DISPLAY MODE 2

Cryostream State: **IDLE**
DISPLAY Lamp: **ON**
PROGRAM Lamp: **OFF**

Description:



Idle phase table

Use the IntelliKnob to scroll through the following information:

Hours	The cumulative time the system has run since manufacture.
Serial#	Controller serial number
Software	The version of the Controller software
Shutdown	This indicates the last reason for shutdown. Options are:
STOP	The STOP button has been pressed

END	The system has been shut down due to a programmed END
PURGE	The system has been shut down due to a programmed PURGE (see *Further Programming on the 700 Series Controller*)
POWER	The power has been switched off at the mains
FLOW	Shutdown due to low flow
TEMP	There has been a large temperature error
SENSOR	This indicates a sensor failure
SINK	Controller overheating
PSU	Power supply overheating
LAST ERR	This stores the last reason for the shutdown but does not include STOP or POWER.

If there has been an unexpected shutdown, the following items are recorded on this list after the LAST ERR:

Set T, Gas T, Gas Heat, Gas Flow, Pressure, EvapT, Heat%, SuctT, Suct Heat, Run Time.

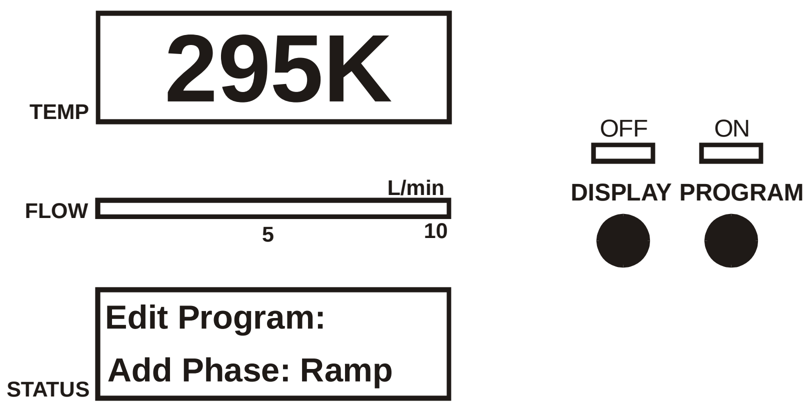
These are recorded to allow the user to diagnose the reason for the shutdown and are stored until there is another erroneous shutdown.

Press DISPLAY or PROGRAM to alter the Display Mode.

3.4.1.3 DISPLAY MODE 3

Cryostream State: **IDLE**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **ON**

Description:



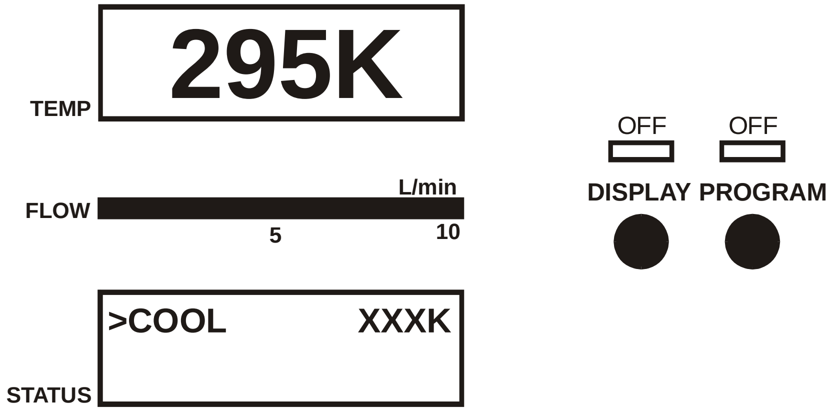
<u>Program mode</u>
This mode allows the user to program the Cryostream as described in Section 4. The list of phases also gives you the option to save or load a program. 'Save Program' will save the current program and 'Load Program' will load the last saved program.

Press DISPLAY or PROGRAM to alter the Display Mode.

3.4.1.4 DISPLAY MODE 4

Cryostream State: **RUNNING**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **OFF**

Description:



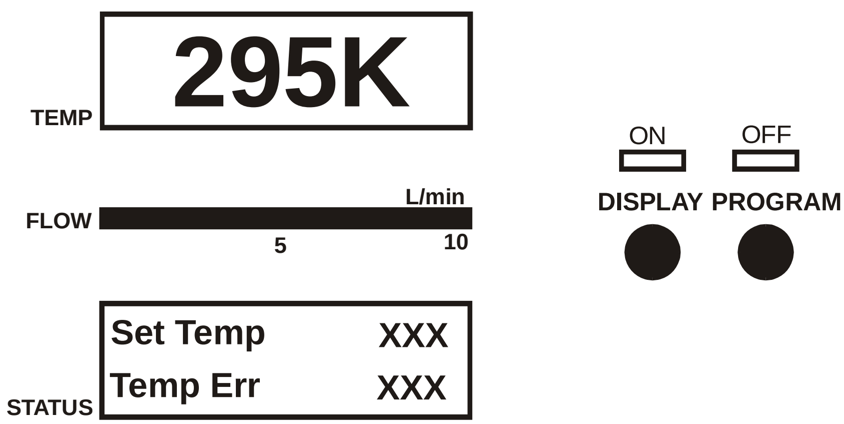
Phase table mode
The Cryostream lists the phases in the current program, with the current phase at the top of the list. The current phase is indicated with a '>'. Use the IntelliKnob to scroll through the list.

Press DISPLAY or PROGRAM to enter the Display Modes 5 & 6.

3.4.1.5 DISPLAY MODE 5

Cryostream State: **RUNNING**
DISPLAY Lamp: **ON**
PROGRAM Lamp: **OFF**

Description:



Running display mode
Use the IntelliKnob to scroll through the following information:

Set Temp	Temperature
Temp Error	Temperature
Run Time	How long system has been running since START was last pressed.
Flow Rate	Gas flow in L/min
Pressure	Back pressure in bars
Gas Heat	The power to the sample heater, expressed as a percentage of full power. The instantaneous value is given and the average is in brackets.
EvapT	Temperature

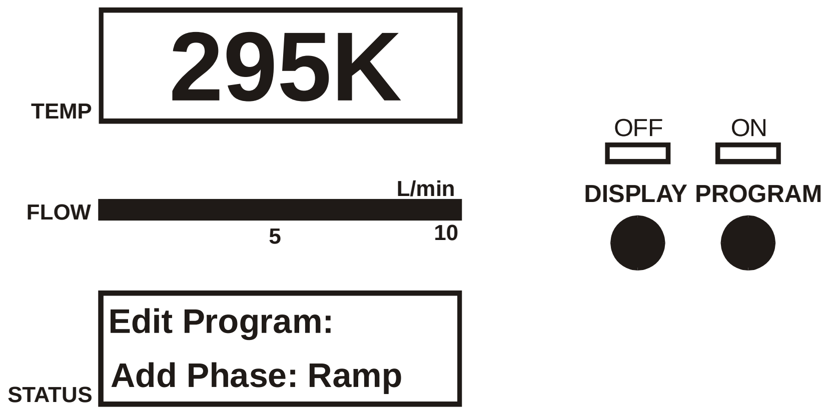
Evap Heat — The power to the evaporator heater, expressed as a percentage of full power. The instantaneous value is given and the average is in brackets.
Evap Shift — The amount in %, the Evap heater has shifted from its ideal operating rate and is used to compensate and then indicate a failing vacuum.
Suct Heat — On or Off
Suct Set Point — Temperature
Suct Temp — Temperature

Press DISPLAY or PROGRAM to alter the Display Mode.

3.4.1.6 DISPLAY MODE 6

Cryostream State: **RUNNING**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **ON**

Description:



Program mode
This mode allows the user to program the Cryostream as described in Section 4. New phases are added at the end of the list of phases. If the system is in a HOLD, press HOLD to begin the next phase.

Press DISPLAY or PROGRAM to alter the Display Mode.

4 Programming the Cryostream

Switch the Cryostream Controller on according to the instructions above and wait for the system to initialise.

4.1 Using the quick-start facility and the COOL function

To cool as quickly as possible simply rotate the IntelliKnob to the appropriate temperature and press START. The Cryostream will remember the last value requested here and store it for the next time the Quick Start facility is used.

NOTE

The system uses the COOL function to get cold as quickly as possible. The COOL function uses a flow rate of 10 L/min to draw the maximum amount of liquid out of the Dewar. This is called **Machine Turbo** and is indicated by the TURBO lamp flashing on and off. Once the system is cold the flow rate will drop to 5 L/min above 90 K. To maintain 10 L/min, press the TURBO button so that lamp remains on permanently.

Once the Cryostream reaches the desired temperature, the Controller will automatically enter a HOLD in the phase table.

With the system now running, press DISPLAY to view *Display Mode 5* discussed above. This displays all the parameters of the system. Alternatively, press PROGRAM to enter *Display Mode 6* and add additional phases to your program (see Section 4.2).

4.2 Further programming of the 700 Series Controller

While the system is idle or running, it is possible to program more detailed phases.

Press PROGRAM (this illuminates the PROGRAM lamp). The STATUS screen then displays the following:

Edit Program:
Add Phase: Ramp

Spin the IntelliKnob to see all the phases. Press ENTER at any time to accept a particular phase.

Here is a list of the possible phases and other parameters that each one requires.

Phase and Description	STATUS Screen Modes
Phase: RAMP <u>Description</u> Change temperature at a controlled rate. When ramping down in temperature, if the selected rate is too fast for the Cryostream to follow, the Controller will automatically enter the RAMP/WAIT mode (this will be indicated on the screen). The effect of this is to stop the ramp in order for the gas temperature to catch up to within 5 K of the gas temperature. The Ramp Rate may be anything between 1 and 360 K/hr.	Edit Program: Add Phase: Ramp Ramp Rate: 120K/hr Final Temp: 100K
Phase: COOL <u>Description</u> COOL is designed to get the system as cold as quickly as possible. It uses a flow rate of 10 L/min to draw the maximum amount of liquid out of the Dewar. This is called **Machine Turbo** and is indicated by the TURBO lamp flashing on and off. Once the system is cold the flow rate will drop to 5 L/min above 90 K. To maintain 10 L/min, press the TURBO button so that the lamp remains on permanently. It is not possible to spin the IntelliKnob above the end temperature of the previous phase or the current gas temperature.	Edit Program: Add Phase: COOL Cool to: 100K
Phase: PLAT <u>Description</u> Maintain temperature fixed for a certain time. The user is prompted to enter a temperature at which to plateau and to specify the plateau's duration. Below 10hr 00min the PLAT function will start to count down in seconds and this will be displayed in the STATUS screen during running.	Edit Program: Add Phase: PLAT Plat Length: 1:00 (hh:mm)
Phase: HOLD <u>Description</u> Maintain temperature fixed indefinitely until the START button is pressed (a programmed HOLD should not be confused with the HOLD button).	Edit Program: Add Phase: HOLD
Phase: PURGE <u>Description</u> This function is designed to warm up the Coldhead as quickly as possible. It applies maximum power to the three heaters in the Coldhead to get to 300 K as quickly as possible. The PURGE is replaced by a SOAKING as the heaters run for a further 10 minutes.	Edit Program: Add Phase: PURGE

Phase: END

<u>Description</u>

System shutdown. You are asked to enter a Ramp Rate back to a final temperature of 300 K and then the system is shut down. This is the controlled way to finish an experiment and should be used whenever possible.

Once an END function has been programmed, it is not possible to enter any more phases. The only options available are to load or save a program or delete the last phase.

Edit Program:
Add Phase: End

Ramp Rate:
120K/hr

Delete Phase

<u>Description</u>

To delete a phase at any time, in Program Mode, spin the IntelliKnob to 'Delete Phase' and press ENTER. This will delete the last phase entered. If the system happens to be executing this last phase, the phase will be replaced by a HOLD.

Edit Program:
Delete Phase

Load Program / Save Program

<u>Description</u>

While the system is idle, it is possible to load or save a program. Loading a Program simply loads the last saved program.

Edit Program:
Load Program

Edit Program:
Save Program

NOTE

Turning the IntelliKnob also offers the option to load a program or save the current program. This is only possible when the system is idle.

Press ENTER on completing each screen. To cancel programming at any time, press PROGRAM or Display (the PROGRAM lamp will go out).

Once the phases have been entered, press START to begin the first phase in the Phase Table. The Controller will automatically enter a HOLD phase at the end of the program if one has not already been programmed. If the system is running and already in a HOLD phase, pressing the HOLD button will pass to the next instruction in the Phase Table.

4.2.1 Phase table

Press PROGRAM at any time during running to look at the phase table and enter more phases. This will enter *Display Mode 6* but will not give the option of loading or saving a program while the system is running.

If there is a list of phases longer than the screen in the phase table, this will be indicated by small characters on the left of the STATUS screen pointing up or down.

↑ ↓	This indicates it is possible to scroll up and down through the phases using the IntelliKnob.
↓	This indicates it is possible to only scroll down through the phases using the IntelliKnob.
↑ ⊥	This indicates that the end of the Phase Table has been reached and it is only possible to scroll up through the phases using the IntelliKnob.
⊤	This indicates that the top of the Phase Table has been reached.
>	This indicates the current programme running.

Table 3 – Phase table

4.2.2 Variable gas flow and the TURBO button

Cryostream offers two gas flow settings: 'Normal' and 'Turbo', selected using the TURBO button and indicated by the TURBO lamp. For temperatures between 90 and 310 K these correspond to flow rates of 5 and 10 L/min respectively. Below 90 K the Normal flow rate is steadily increased from 5 L/min at 90 K to 10 L/min at 80 K in order to maintain temperature. Above 310 K, the Turbo flow rate is 5 L/min, identical to the Normal flow. This is illustrated in Figure 7.

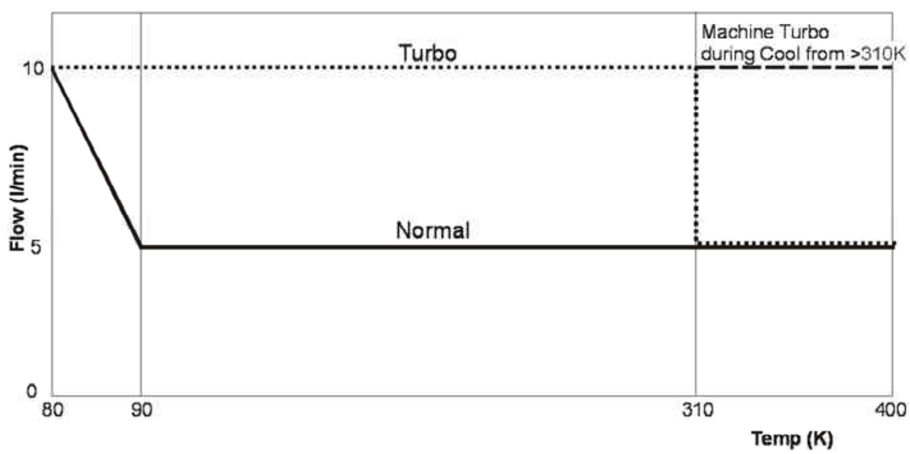


Figure 7 – Variable gas flow

In addition to the Normal and Turbo settings, Cryostream enforces a 'Machine Turbo' during COOL phases. This corresponds to a flow rate of 10 L/min and is indicated by a flashing TURBO lamp.

4.2.3 HOLD and 'Un-HOLD'

A program can be paused at any time using the HOLD button, this will illuminate the HOLD lamp. To continue the program simply press the HOLD button at any time and the HOLD lamp will go out.

It is also possible to release the HOLD phase by pressing the START button.

4.2.4 Alarm Conditions

The 700 Series Controller has a number of safety features. If there is an issue with the system, an alarm condition is indicated by an illuminated ALARM lamp and a warning will appear on the bottom screen of the Controller. The following list describes all the warnings stored by the Controller.

Temp Warning

If the temperature error has reached 10 K the Controller will indicate a warning but will not shut down.

Pressure Warning

If a blockage forms inside the Coldhead, the Controller will sense the back pressure and trigger a pressure warning. To remove this ice blockage, restart the system and program a PURGE. A PURGE will heat up the system as quickly as possible.

Poor Vacuum?

Under certain circumstances, it's possible for the Controller to detect a poor vacuum by comparing certain heater and temperature values at certain times. It is recommended that the vacuum is pumped down if this warning appears. See section 7.

Self-Check Fail

During the initialisation, the Controller checks a variety of parameters to make sure that everything is connected properly and that there is continuity in all parts of the system. Try restarting the Controller a number of times to see if the problem persists. If it does, contact Oxford Cryosystems.

Flow Rate Fail

As the Controller is controlling the flow of gas through the system it will indicate if there is a gas flow problem. This could be due to a blockage or restriction, no source gas or an outward leak of source gas.

Temp Control Err

If the temperature error has reached 25 K and the Controller reads this value five times from the system, the Controller will indicate a warning and shut down.

Temp Reading Err

The Controller has received a nonsense reading from the temperature sensors.

Suct Temp Err

The Cryostream has a temperature sensor at the SUCT connector on the Coldhead. If this error message is displayed on the Controller, contact Oxford Cryosystems.

Sensor Fail

If the Controller receives extreme values from the sensors, it will try to reset them. If the sensors fail to reset after five attempts, the Controller will shut down with this error.

Brown Out

If there is a brief interruption in the electrical supply to the Controller, the Controller will indicate a 'Brown Out' has occurred. The Controller will continue to function normally.

Sink Overheat

If the Controller overheats, there will be a Sink Overheat warning. This is often due to the covering of the fan on the underside of the Controller.

PSU Overheat

If the Controller overheats, there could be a PSU Overheat warning. This is often due to the covering of the fan on the underside of the Controller.

Power Loss

When the power to a Controller is cut, the Controller will report a Power Loss error in the diagnostic screen when it is restarted.

NOTE

When there is a warning or if the system shuts down, the ALARM lamp will flash quickly and the buzzer will sound. See the diagnostics by pressing DISPLAY and report these to Oxford Cryosystems.

4.3 Safety features during power failures

The 700 Series Controller is designed to protect itself and the sample during power interruptions.

It is possible for the Controller to maintain gas flow and not reset the Controller during electrical interruption indicated by the term 'Brown-Out' of between 0-2 seconds. If a 'Brown-Out' is detected, this is indicated on the screen.

4.4 Refilling the Dewar vessel from a storage vessel

Although it is possible to simply refill the Cryostream Dewar by pouring liquid nitrogen directly from a secondary Dewar, it is recommended that a self-pressurised storage vessel be used where possible.

The Cryostream Dewar vessel can be refilled at any time. Although the Cryostream operates at atmospheric pressure, it is important that the liquid nitrogen is delivered into the Cryostream Dewar vessel correctly.

1. Make sure the pressure in the storage tank being used to refill the Cryostream Dewar vessel is no greater that 2 bar.
2. The transfer line from the storage tank to the Cryostream Dewar vessel is less than 2 metres long.
3. Vent the transfer line to begin with to avoid spraying warm air from the transfer line in the Cryostream Dewar vessel. This will evaporate the liquid from the Dewar.

4. The end of the transfer line should not be submerged into the liquid nitrogen in the Cryostream Dewar vessel.
5. Any large storage vessel should be grounded (earthed) before using it to top up the Cryostream Dewar vessel. This avoids any large static discharges from the vessel in to the Cryostream Controller.

4.5 How to shut down the Cryostream

To shut down the Cryostream correctly, the user should program in an END phase into the phase table. The Cryostream will ramp to 300 K and will then shut down. This will allow the nozzle to become warm, and thus help prevent moisture from the air migrating up the nozzle.

In the case of an emergency, press the red STOP button.

Once the Cryostream has shut down it can simply be reprogrammed to start the system up again. **It is not necessary to switch the system off**. Simply press START to re-initialise the system.

After the Cryostream has been switched off it is advisable to leave the liquid nitrogen leg in the LN2 Dewar (e.g. until the next day) so that the Cryopump can maintain a good vacuum while the heat exchanger warms up gradually. This will prevent condensation or ice forming on the grey Coldhead that may cause nuisance to surrounding equipment.

4.5.1 Cryostream shut downs

The 700 Series Controller has been designed to fully protect the Cryostream under its normal mode of operation as outlined in this manual. The Controller will shut down the Cryostream if:

- The Gas Temp or registers an error greater than ±25 K.
- Gas flow drops below 2 L/min.
- The Controller or power supply overheats.
- A temperature sensor (Gas temperature, Evaporator, or SUCT temperature) fault occurs.
- External electrical noise (from a rotating anode for example) is too great to allow accurate temperature readings to be taken.

In each case the run time variables are saved (see *Display Mode 3*) and a suitable error message is displayed.

In the case of an unexpected shut down, record the reason for the shutdown as indicated on the STATUS screen and press START to have the option to reprogram it. Pressing START will again, reinitialise the Controller. To see a full list of diagnostics at the time of shut down, press DISPLAY which will take you into *Display Mode 2*. Use the IntelliKnob to scroll through the 'Last Shutdown Diagnostics' listed after the 'Last Err' entry.

If the shutdown is due to the gas flow dropping below 2 L/min, the Controller will automatically enter and execute a PURGE phase.

5 Running the Cryostream with Cryopad

Cryopad is a PC program which allows remote monitoring and control of any 700 series Oxford Cryosystems device. This includes the 700 series Cryostream / Plus, Cobra / Plus, PheniX and N-HeliX systems.

5.1 Installing Cryopad

Install Cryopad from the CD also supplied with the Cryostream or download from the Oxford Cryosystems website, www.oxcryo.com/software/cryopad. If you experience problems with the web installer please install the Microsoft Visual C++ Redistributable Package as explained on the website.

5.2 Using Cryopad to run the Cryostream

With the controller switched off connect a COM port from your PC to the port labelled SERIAL on the back of the Controller. **This connection can be made with any standard M-F serial cable**. Now turn on the Controller and start Cryopad by selecting from the Oxford Cryosystems group in the Start Menu, or else by double-clicking the Cryopad logo on your desktop.

5.2.1 Connecting using the Settings page

The first time you use Cryopad you will need to select a COM port using the Settings page. If you know which COM port you are using then select it from the **Connect Using Port** menu. If you are using a non-standard COM port you may type its name directly here. Alternatively click the **Search…** button to display the Search dialog, which may be used at any time to scan your computer's COM ports for compatible devices.

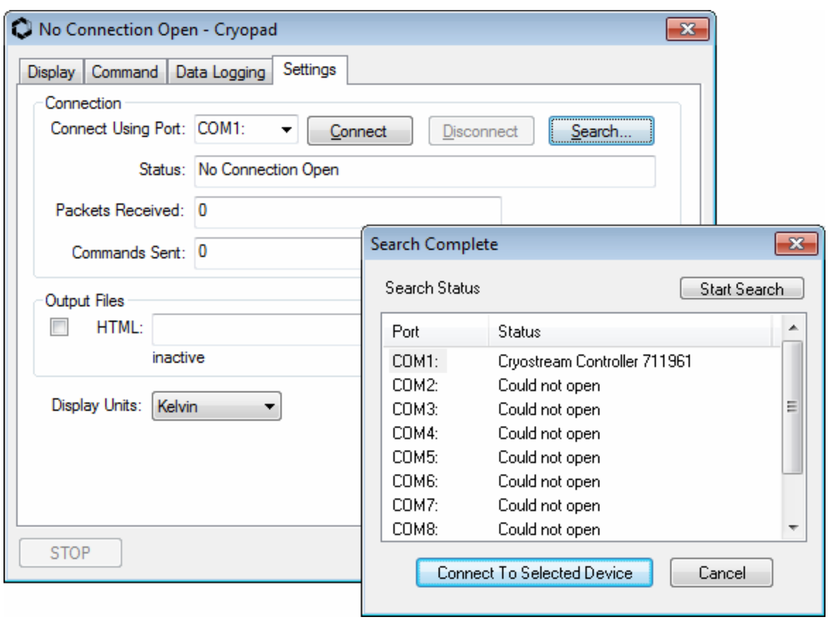


Figure 8 – Cryopad Settings page and Search dialog

The next time you run Cryopad your chosen COM port will be opened automatically, and a connection will be established as soon as a compatible device is detected. Should you need to change the COM port, switch to the Settings page, press Disconnect and repeat the above procedure. Should you wish to connect more than one device, run a new copy of Cryopad for each device and connect each one as described above.

5.2.2 The Display page

Once a connection has been established the Display page shows the live status of the device. The table below indicates the meaning of the various quantities displayed. After half an hour or so when the device has reached its normal operating values all the indicators will appear green.

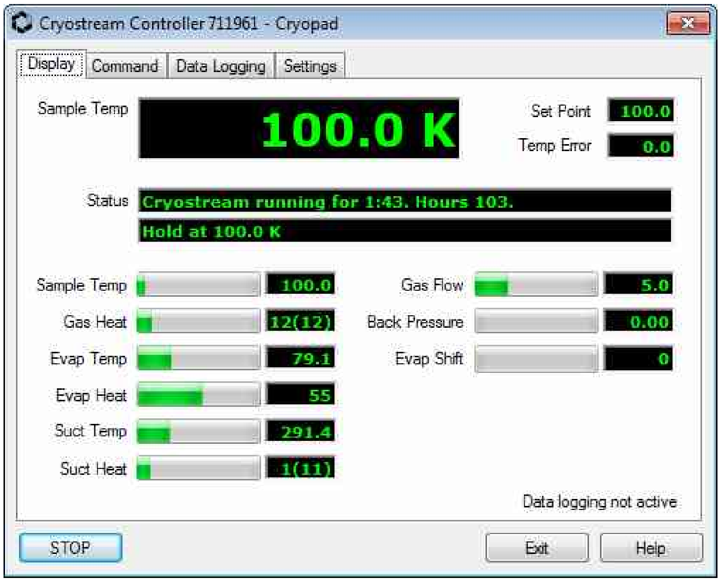


Figure 9 – Cryopad Display page

Data	**Explanation**
Sample Temp	The gas temperature at the crystal position.
Set Point	The temperature to which Cryostream is controlling.
Temp Error	The difference between the Sample Temp and the Set Temp, except in a Cool phase, in which Temp Error is zero.
Status	Two lines of information indicating the current status of Cryostream. Any errors or warnings raised by Cryostream will be displayed here.
Gas Heat	The power to the sample heater, expressed as a percentage of full power.
Evap Temp	The temperature measured by the evaporator sensor.
Evap Heat	The power to the evaporator heater, expressed as a percentage of full power.
Suct Temp	The temperature measured by the suct sensor.
Suct Heat	The power to the suct heater, expressed as a percentage of full power.
Gas Flow	The gas flow in litres / minute. The letter T indicates that Cryostream is in Turbo mode.
Back Pressure	The back pressure in the gas line, measured in bar.
Evap Shift	A parameter indicating the amount by which the evaporator heater power has been reduced to allow very low temperatures to be attained.

5.2.3 The Command page

The Command page allows commands to be sent to your Cryostream exactly as if they were entered using the Controller. Commands sent in this way will immediately overwrite the contents of the Controller's Phase table. Refer to section 4 above for details of the commands.

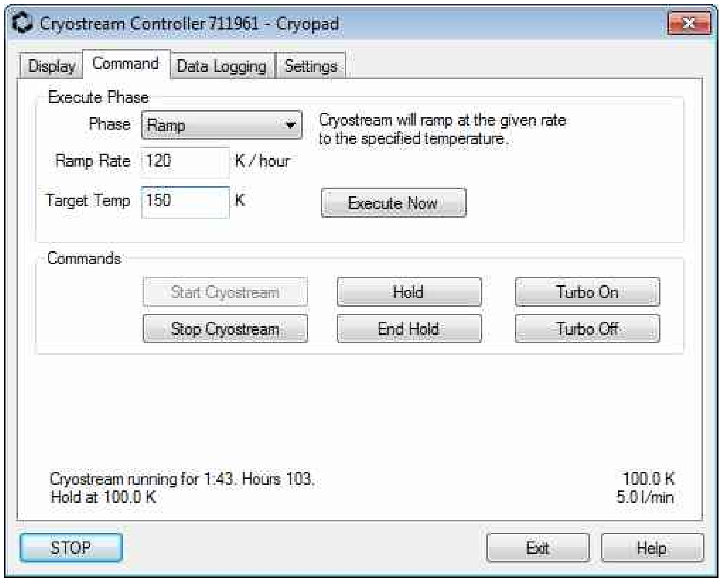


Figure 10 – Cryopad Command page

5.2.4 The Data Logging page

Cryopad allows data to be logged to a tab-delimited text file suitable for use in Excel or similar programs. Choose a file to which the data will be logged using the Log File item, and select the quantities of interest using the check boxes. The Interval item allows you to select the interval in seconds at which the data are logged. For monitoring purposes a 60 s interval is suitable whereas for diagnosing problems an interval of 1 s provides the most information but will produce a larger log file. Check the Logging Active box to commence logging.

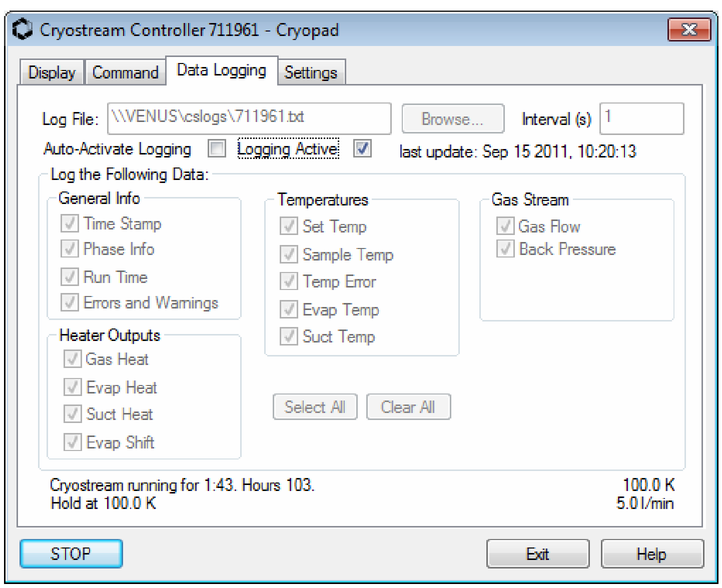


Figure 11 – Cryopad Data Logging page

6 Measuring the true crystal temperature

6.1 How the Cryostream measures the crystal temperature

In contrast to other cooling systems and indeed previous versions of the Cryostream, the Cryostream displays the gas temperature at the position of the crystal, rather than a point within the Coldhead. Oxford Cryosystems has established a method of mapping the true temperature at 5 mm from the end of the nozzle. Because of the superior laminar flow of the Cryostream gas stream, one can place the sample up to 10 mm from the end of the nozzle.

The correction which needs to be applied is a function of gas flow, which means that switching between Normal and Turbo flows will cause a genuine change in the crystal temperature. The Cryostream will compensate for this change, but nevertheless unnecessary changes in the flow rate should be avoided if small changes in gas temperature at the crystal are not desirable.

6.2 How to measure the crystal temperature at your crystal position

NOTE
We do not recommend measuring the temperature with a thermocouple placed in the stream.

If it is important to know the actual temperature at the position of your crystal for an experiment, you can always check it. In the heterogeneous environment of a narrow cold gas stream, there are several factors that lead to spurious voltages on the thermocouple, creating errors in apparent temperature of possibly tens of degrees! For instance, conduction of heat down the wires creates a heat leak. At the point of entry of the thermocouple wires into the stream a cold junction is formed whose temperature is much lower than the room temperature assumed by the Controller, thus making the measured temperature *appear* to be much higher than indicated. Also, the sharp temperature change at the interface between the cold stream and the surrounding warm air can induce stresses into the thermocouple wires and then generate spurious EMFs. We believe that the only satisfactory way to find the error in absolute temperature at the crystal position is to calibrate with a sample that undergoes a known phase transition or change of state. For instance, we have found that the low temperature phase transition in the langbeinite $(NH_4)_2Cd_2(SO_4)_3$ was observed from intensity measurements to be in the range 88-89 K (established elsewhere to be at 88 K). Similarly, lattice parameter measurements of sodium ammonium tartrate tetrahydrate (Rochelle Salt) gave a transition temperature in agreement to within 0.5 K of the published value of 109 K. A most useful compilation of transitions in hundreds of crystals has been published by P. Tomaszewski (*Phase Transitions*, **38**, 127).

7 Cryostream user maintenance

The Cryostream has been designed to be as easy to use as possible and should run without the need for constant attention, once the user is accustomed to the system. However, some parts of the system will eventually require maintenance, with the following maximum service intervals.

Procedure	Maximum Service Interval
Pumpdown of Vacuum (See Section 7.1)[1]	2 Years
Replacement of Pump Diaphragm[2]	12,000 Hours
Replacement of Line Drier (See Section 2.6)[3]	12,000 Hours
Service of Dry Air Unit[4]	15,000 Hours

Table 4 – Cryostream Service Intervals

1. If the outer case of the Cryostream Coldhead becomes wet or frosty, this probably indicates a loss of vacuum. The remedy is to pump out the system as described in Section 7.1.

2. The Cryostream gas pump unit contains two rubber diaphragms. Possible symptoms of a diaphragm failure can be found in the Troubleshooting Guide. Do not rush to change this diaphragm as failure to replace it properly can result in the introduction of gas leaks. Contact Oxford Cryosystems for more information.

3. Regular maintenance of the line drier units is recommended, as they will become saturated with water from the nitrogen supply

4. The Counter on the front of the AD51 indicates when this system needs servicing. If demand on the AD51 is high, plan to service the AD51 before it fails at a time that is convenient. Instructions for servicing the AD51 are in its manual. An AD51 Service Kit can be obtained from Oxford Cryosystems or a local Cryostream agent.

7.1 Pumping down the vacuum space in the Cryostream

From time to time you may need to re-pump the vacuum insulation space of the Cryostream Coldhead. This will be apparent when the outside of the Coldhead and transfer line becomes excessively cold or wet all over during operation or the Cryostream is unable to reach the required gas temperature and the Heat% value is zero.

Do not be misled by similar symptoms that are not due to a vacuum problem. For instance, localised condensation or icing of the SUCT line is not likely to be caused by poor vacuum. Similarly, if the liquid nitrogen supply runs low or when the nitrogen leg is removed from the storage vessel the operation of the built-in cryopump is defeated and the Coldhead may become cold or damp externally.

It has been established that the Cryostream vacuum can be re-pumped using a good rotary vacuum pump (with air ballast valve), a Pirani gauge and the CRH25 Reactivation Heater. It is not necessary to use a diffusion pump or turbomolecular pump – in fact, backstreaming of diffusion pump fluid could well contaminate the Coldhead.

The correct procedure is as follows:

1. Determine that the unit really does require re-pumping as described above (see Troubleshooting Guide or contact Oxford Cryosystems or your local agent if you are unsure).

2. Switch the Cryostream off and leave it standing for 24 hours. This will allow it to warm up internally. Alternatively chose the PURGE option on the Controller which will only take 1 hour 45 minutes.
3. Take the rigid section of the transfer line out of the storage vessel. Leave the rigid leg to warm up for an hour and then dry it carefully.
4. Place the end of the nitrogen leg into the hole in the hot block of the **CRH25** heater. Ensure that nothing else can come into contact with the heater to cause damage. **Do not switch the heater on at this stage.**
5. Connect a good rotary pump (preferably 2-stage) to the Cryostream Coldhead pump-out port using the pumping Adaptor supplied (see Figure 12). Screw the adaptor onto the pump-out port positioned at the top of the rigid leg of the transfer line. **Do not withdraw the sealing plug at this stage.**

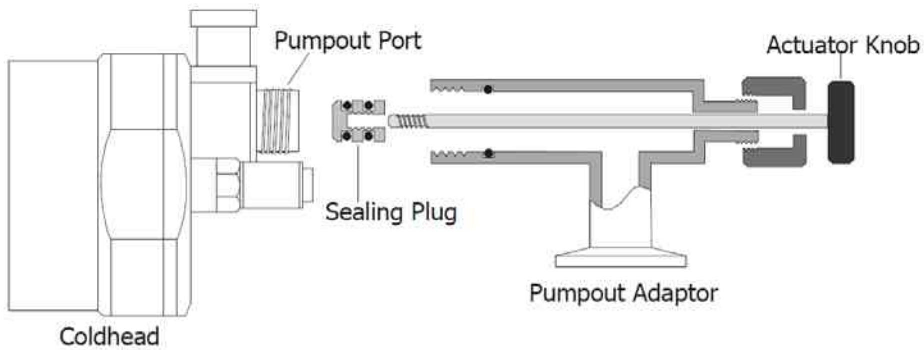


Figure 12 – Attaching the pumping adaptor

6. Start the rotary pump and ensure that a pressure of 0.1 mbar can be obtained up to the pumping adaptor. It may be necessary to run the pump with its air ballast valve open for about 30 minutes.
7. When the rotary pump pressure is 0.1 mbar, use the pumping adaptor actuator knob to locate and withdraw the sealing plug very slowly to avoid a rush of gas. An extra valve in the pumping line would help in this operation.
8. Wait until the pressure falls towards 0.1mbar (this may take 60 minutes). If necessary, use the rotary pump air ballast again. If you cannot obtain a sufficiently low pressure at this stage you may have a leak (or bad pump) which should be investigated. Contact your Cryostream supplier if all else fails.
9. When a pressure near to 0.1 mbar is obtained, the CRH25 reactivation heater can be switched on. The heater raises the temperature of the cryopump situated in the bottom of the nitrogen leg to high temperature in order to drive off water and other condensable vapours. This is likely to cause the vacuum space pressure to rise and it may well be necessary to use the rotary pump air ballast yet again to purge the pump of these condensables.
10. The vacuum should be pumped and baked for an absolute minimum of 16 hours, and preferably for up to 72 hours where possible. The final pressure should be 0.1 mbar (or better) with a 2-stage rotary pump. You may well achieve 0.01 mbar but do not worry too much as Pirani gauges are not always very accurate.
11. To finish pumping and baking, the sealing plug should be pushed back in using the actuator knob of the pumping adaptor. **Note: This must be done whilst the CRH25 heater is still hot.**
12. Switch the CRH25 heater off immediately and allow to cool. Unscrew the actuator knob from the sealing plug and remove the pumping port adaptor. Replace the pumping port cover.

8 Liquid and gaseous nitrogen safety sheet

8.1 General

These safety points are a guideline to outline the potential hazards and procedures involved in the handling of liquid or gaseous nitrogen. Anyone handling liquid or gaseous nitrogen should first inform their departmental or laboratory safety advisor and receive advice about local safety procedures.

All users are requested to read this safety sheet before handling the Cryostream. Oxford Cryosystems accept no responsibility for injury or damage caused by the mishandling of liquid or gaseous nitrogen.

8.1.1 General properties

- Gaseous nitrogen is colourless, odourless and tasteless and is slightly lighter than air at equal temperatures; cold nitrogen vapour is, however, denser than atmospheric air.
- Liquid nitrogen is odourless, colourless and boils at -195.8°C. One volume of liquid nitrogen gives approximately 700 volumes of gas at ambient conditions.
- Nitrogen is not flammable. It is chemically inert, except at high temperatures and pressures. Its volume concentration in air is 78%.
- Liquid and cold gaseous nitrogen can cause severe burns or frostbite when in contact with the skin or respiratory tract.
- Gaseous and liquid nitrogen is non-corrosive.
- Nitrogen does not support life and acts as an asphyxiant.
- Nitrogen is intrinsically non-toxic.

8.2 Fire and explosion hazards

Gaseous and liquid nitrogen are non-flammable and do not themselves, constitute a fire or explosion risk. However, both gaseous and liquid nitrogen are normally stored under pressure and the storage vessels whether gas cylinders or liquid tanks, should not be located in areas where there is a high risk of fire or where they may normally be exposed to excessive heat.

8.3 Health hazards

8.3.1 Asphyxia

Nitrogen, although non-toxic, can constitute an asphyxiation hazard through the displacement of the oxygen in the atmosphere. Nitrogen gas or oxygen depletion is not detectable by the normal human senses.

Oxygen is necessary to support life and its volume concentration is the atmosphere is 21%. At normal atmospheric pressure persons may be exposed to oxygen concentrations of 18% or even less, without adverse effects. However, the response of individuals to oxygen deprivation varies appreciably. The minimum oxygen content of breathing atmospheres should be 18% by volume but to ensure a wider margin of operational safety it is recommended that persons are not exposed to atmospheres in which the oxygen concentration is, or may become, less than 20% by volume.

Symptoms of oxygen deprivation, such as increased pulse and rate of breathing, fatigue, and abnormal perceptions or responses, may be apparent at an oxygen concentration of 16%.

Permanent brain damage or death may arise from breathing atmospheres containing less than 10% oxygen. Initial symptoms will include nausea, vomiting and gasping respiration. Persons exposed to

such atmospheres may be unable to help themselves or warn others of their predicament. The symptoms are an inadequate warning of the hazard.

WARNING
Breathing a pure nitrogen atmosphere will produce immediate loss of consciousness and almost immediate death.

8.3.2 Cold burns

Liquid and cold nitrogen vapours or gases can produce effects on the skin similar to a burn. Naked parts of the body coming into contact with un-insulated parts of equipment may also stick fast (as all available moisture is frozen) and the flesh may be torn on removal.

8.3.3 Frostbite

Severe or prolonged exposure to cold nitrogen vapour or gases can cause frostbite. Local pain usually gives warning of freezing but sometimes no pain is experienced. Frozen tissues are painless and appear waxy with a pallid yellowish colour. Thawing of the frozen tissues can cause intensive pain. Shock may also occur if the burns are at all extensive.

8.3.4 Effect of cold on lungs

Prolonged breathing of extremely cold atmospheres may damage the lungs.

8.3.5 Hypothermia

Low environmental temperatures can cause hypothermia and all persons at risk should wear warm clothing. Hypothermia is possible in any environmental temperature below 10ºC but susceptibility depends on time, temperature and the individual. Older persons are more likely to be affected. Individuals suffering from hypothermia may find that their physical and mental reactions are adversely affected.

8.4 Precautions

8.4.1 Operations and maintenance

It is essential that operations involving the use of gaseous or liquid nitrogen particularly where large quantities are used are conducted in well-ventilated areas to prevent the formation of oxygen deficient atmospheres.

Ideally, nitrogen should be vented into the open air well away from areas frequented by personnel. It should never be released or vented into enclosed areas or buildings where the ventilation is inadequate. Cold nitrogen vapours are denser than air and can accumulate in low lying areas such as pits and trenches.

Where large spills of liquid nitrogen occur, a fog forms in the vicinity of the spill caused by the condensation of water vapour in the surrounding air. The fog, in addition to severely reducing visibility may contain oxygen concentrations appreciably lower than that of the air presenting a local asphyxiation hazard.

8.4.2 Personnel protection

Persons handling equipment in service with liquid nitrogen should wear protective face shields, loose fitting gauntlets and safety footwear.

8.4.3 Emergencies

In the event of an accident or emergency the instructions below should be implemented without delay.

8.4.4 Asphyxiation

Persons showing symptoms of oxygen deprivation should be moved immediately to a normal atmosphere. Persons who are unconscious or not breathing must receive immediate first aid. Medical assistance should be summoned without delay. First aid measures included inspection of the victim's airway for obstruction, artificial respiration and simultaneous administration of oxygen. **These procedures should only be carried out by a trained first aid staff**. The victim should be kept warm and resting.

It is important that the personnel carrying out rescue operations should minimise the risk to themselves.

8.4.5 Treatment of cold burns and frostbite

Cold burns should receive medical attention as quickly as possible. However, such injuries are not an everyday occurrence and doctors, hospital staff or works first aid personnel may not be aware of the basic methods of treatment. The following notes describe the first aid treatment and recommended advice for further treatment to be given by a medical practitioner or a hospital.

8.5 First Aid

In severe cases summon medical attention immediately. Flush affected areas of skin with copious quantities of tepid water to reduce freezing of tissue. Loosen any clothing that may restrict blood circulation. Move the victim to a warm place but not to a hot environment and do not apply direct heat to the affected parts. Every effort should be made to protect frozen parts from infection and further injury. Dry, sterilised bulky dressings may be used but should not be applied so tightly that blood circulation is restricted.

8.5.1 Treatment by Medical Practitioner or Hospital

1. Remove any clothing that may constrict the circulation to the frozen area. Remove patient to sick bay or hospital.
2. Immediately place the part of the body exposed to the cryogenic material in a water bath which has a temperature of not less than 40°C but no more than 45°C. **Never use dry heat or hot water**. Temperatures in excess of 45°C will superimpose a burn upon the frozen tissue.
3. If there has been a massive exposure to the super cooled material so that the general body temperature is depressed, the patient must be re-warmed gradually. Shock may occur during re-warming, especially if this is rapid.
4. Frozen tissues are painless and appear waxy with a pallid yellowish colour. They become painful, swollen and very prone to infection when thawed. Therefore, do not re-warm rapidly if the accident occurs in the field and the patient cannot be transported to hospital immediately. Thawing may take from 15-60 minutes and should be continued until the blue, pale colour of the skin turns to pink or red. Morphine, or some potent analgesic, is required to control the pain during thawing and should be administered under professional medical supervision.
5. If the frozen part of the body has thawed by the time medical attention has been obtained, do not re-warm. Under these circumstances cover the area with dry sterile dressings with a large bulky protective covering.
6. Administer a tetanus booster after hospitalisation.

8.5.2 Hypothermia

Persons suspected to be suffering from hypothermia should be wrapped in blankets and moved to a warm place. Slow restoration of temperature in necessary and forms of locally applied heat should not be used. Summon medical attention.

8.5.3 Liquid nitrogen spillage

If large spills of liquid nitrogen spillage occur, large quantities of water should be used to increase the rate of liquid vaporisation.

9 Cryostream troubleshooting guide

9.1 Very important guidelines for using this document

1. This guide is designed for operators responsible for looking after Cryostream.
2. This guide is not designed to cover every technical eventuality but to provide the correct interpretation of, and solution to, a variety of common symptoms. As a user, symptoms may arise that are not covered here. If, at any time, you are unsure of the cause of the Cryostream problem, contact your local agent or Oxford Cryosystems directly.
3. If you experience a shutdown or unusual behaviour from your system, please record as much information as possible and any physical symptoms you feel are a concern. Then contact Oxford Cryosystems or your local agent.
4. **Do not** rush into changing components or fixing something until you have spoken to Oxford Cryosystems (remember technical support and advice are free of charge) or your local agent. Changing components can **very often** create more problems and mask the original fault.

9.2 Problems and solutions

9.2.1 Condensation and/or ice covering the outside of the Coldhead or the flexible transfer line

Cause

Condensation and/or ice over the outside of the Cryostream transfer line or Coldhead indicates a loss of vacuum. This should not be confused with localised spots of ice or condensation. Remember, it is impossible to lose a vacuum from one small area of vacuum space! This loss of vacuum can be for two reasons:

1. Natural out-gassing over a period time depending on the physical treatment of the Coldhead.
2. Vacuum leak at an internal or external joint or cracked flexible line, although this is rare.

Solution

Consult the instruction manual on re-pumping the vacuum.

If it is believed there is a leaky joint or cracked flexible hose, contact Oxford Cryosystems or your local agent as this is not user serviceable.

Associated symptoms

- Inability to reach low temperatures and the base temperature begins to rise
- Inability to reach low temperatures
- Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

9.2.2 Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

Cause

This is caused by a failing of the vacuum insulation in the Coldhead and is determined by the Controller diagnostics. This is done by analysing the power to the various heaters in the Coldhead:

1. Natural out-gassing over a period time depending on the physical treatment of the Coldhead.
2. Vacuum leak at an internal or external joint or cracked flexible line, although this is rare.

Solution
Consult the instruction manual on re-pumping the vacuum.

If it is believed there is a leaky joint or cracked flexible hose, contact Oxford Cryosystems or your local agent as this is not user serviceable.

Associated symptoms

- Inability to reach low temperatures and the base temperature begins to rise
- Inability to reach low temperatures

9.2.3 Localised ice spot on the flexible line

Cause
This is due to the flexible line being bent too sharply beyond its 200 mm minimum radius so that the transfer capillary inside the flexible vacuum jacket touches the wall of the vacuum jacket.

Solution
The Cryostream will continue to run happily like this, but it may be worth contacting Oxford Cryosystems or your local agent. Try to increase the bend radius at the point where the ice spot occurs.

9.2.4 Localised ice formation around the neck of the Dewar and the Cryostream leg

Cause
If the Dewar is open to the atmosphere, it is common for ice to build up at the interface between the warm air and the cold gas eminating from the Dewar. This ice can fall into the Dewar and contaminate the nitrogen as its concentration builds up.

Solution
Find a way to cover the Dewar opening. This can be done quite comprehensively but **be sure not to seal the Dewar**. Simply plugging the Dewar with a cloth will prevent most atmospheric moisture getting into the Dewar, but more elaborate setups involve clamping a bung on to the top of the Dewar and then drilling holes for the Cryostream leg, venting and refilling. If the Dewar is sealed up, a vent hole is **very** important otherwise the Cryostream will **not** work.

Associated symptoms

- Cryostream shutdown due to low flow after a few seconds of running
- Cryostream shutdown due to low flow - no explicable reason

9.2.5 Inability to reach low temperatures

Cause
If the gas temperature will not drop below a certain temperature, (for example, the programmed gas temperature is 100 K and the Cryostream will only reach 105 K but is stable and not rising), or has been rising over a period of time, the vacuum may be failing. The 700 Series is designed to monitor vacuum failure as it has control over the heat in the system. If there are vacuum problems when trying to run at 100 K, the Controller Alarm will ring and the vacuum warning will appear on the screen.

Solution
If the base gas temperature that the Cryostream reaches continues to rise, it is likely that the vacuum is degrading and will require re-pumping. See the manual for this procedure.

Associated symptoms

- Condensation and/or ice covering the outside of the Coldhead or the flexible transfer line
- Inability to reach low temperatures and the base temperature begins to rise
- Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

9.2.6 Inability to reach low temperatures and the base temperature begins to rise

Cause

These symptoms indicate a loss of vacuum due to the natural out gassing over a period time depending on the physical treatment of the Coldhead. At lower temperatures, the Vacuum Warning Alarm should sound. At higher temperatures, a temperature error is more likely.

Solution

Re-pump the vacuum.

Associated symptoms

- Condensation and/or ice covering the outside of the Coldhead or the flexible transfer line
- Inability to reach low temperatures
- Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

9.2.7 Ice formation on the sample

Cause

Ice formation on the sample can begin at the point of flash cooling the sample or it can build up over time to eventually cover the sample and thus ruin the diffraction image.

NOTE

Ice on the sample does not come from the nitrogen gas travelling down the nozzle. Nitrogen gas from the Cryostream is very dry (circa 0.1 ppm of water vapour).

Ice on the thin film supporting the crystal in the loop can arise from a number of sources.

1. Insufficient cryoprotection of the buffer solution.
   a. Too much mother liquor results in dilution of the cryoprotectant to the point where it is no longer adequate.
   b. A thick film around the crystal may result in a larger thermal mass that must then be cooled.
2. Rate of flash cooling is too slow.
3. The sample is too far away from the nozzle or not aligned in the centre of the cold stream. The cold stream and the dry air stream mix and draw in atmospheric moisture that is frozen out on the sample.
4. The loop is unclean. Any particles on the loop will propagate ice formation.
5. A wet dry air supply or a disturbance of the laminar flow system due to drafts in your laboratory or an oversized sample mount (ie capillary or pin is too thick).

6. It is important that the velocities of the two gases are the same. If they are grossly unmatched, atmospheric moisture will encroach the streams and cause ice to build up. A true laminar flow will prevent attack from atmospheric moisture.

Solution

To increase the effectiveness of the cryoprotectant, increase its concentration.

Position the nozzle as close to the sample as possible without affecting the path of the x-rays or casting an image on to the detector. The ideal position is inside the first 6 mm from the end of the nozzle and the centre 2 mm cross section. Be sure to clean the loop before use as ice build up will only compound the problem.

Check the laboratory for drafts.The most likely cause of turbulence is an air conditioning unit, a cooling fan from an x-ray generator or the rotating anode generator. Create a screen between the source of the draft and your cold stream. This will greatly reduce the turbulence. If you are unsure of the source of the draft, try the Flashlight Test.

Try adjusting the flow of the outer dry gas stream. 12-13 litres per minute is normally fine, but when the air is more turbulent, try turning the outer stream flow rate up to a maximum of 15 litres per minute; this can often cure the problem. (See Flashlight Test, Section 9.2.15)

If the icing persists and there is also a concentric build up of ice on the nozzle, the most likely cause is a wet dry air supply. If you have an Oxford Cryosystems Dry Air Unit, change the Compressor Filter Delivery Element. If the icing persists, contact Oxford Cryosystems or your local agent about a Dry Air Unit service.

Associated symptoms

- Concentric formation of ice around the nozzle.

9.2.8 Ice formation on outer edge of the nitrogen gas cold stream nozzle

Cause

The likely cause of ice on one side only of the nitrogen nozzle is a misaligned dry air shroud tube.

Solution

Look up the nozzle of the Cryostream and check to make sure the outer dry air shroud is concentric with the inner nitrogen nozzle. A small misalignment may be corrected by pushing the inner nozzle to one side. The shroud tube is locked into its 26mm diameter mounting bush using a low strength retainer compound. To release the shroud tube, grasp it gently and push to one side to release the retainer bond. Movement of the shroud tube will be limited as it touches the outside of the inner nitrogen nozzle - this prevents the shroud tube from kinking. Once the outer dry air shroud has been removed, refit the shroud tube using a little retainer compound, check that the tube is concentric and allow the retainer to set.

9.2.9 Concentric formation of ice around the nozzle

Cause

This is likely to be wet air from the dry air supply or a high flow rate from the dry air supply. The cold stream requires a dry air shroud of dewpoint -60°C. If the stream is wet, the moisture in the air will freeze onto the nozzle and sample.

Solution
Make sure an unruly student has not turned the flow rate up to 25 L/min!! If the Cryostream utilises an Oxford Cryosystems AD51 Dry Air Unit, change the Compressor Delivery Filter Element; a spare is provided. If the ice persists, contact Oxford Cryosystems or your local agent who will supply a service kit.

Associated Symptoms
Ice formation on the sample

Ice formation on outer edge of the nitrogen gas cold stream nozzle

9.2.10 Positive Gas Temperature Error

Cause
If after months of use, the Cryostream starts to lose its vacuum insulation, then it will struggle to maintain its base temperature and the gas temperature error will increase, positively.

Solution
Re-pump your vacuum insulation in accordance with the instructions in the manual.

Associated Symptoms
Condensation and/or ice covering the outside of the Coldhead or the flexible Transfer Line.

Inability to reach low temperatures and the base temperature begins to rise.

9.2.11 Cryostream shutdown due to low flow after a few seconds of running

Cause
If, after a few seconds of running, the Gas Pump Unit can not generate any gas flow the system will shut down. The most likely cause of this is a restriction in the transfer line from the Dewar caused by a solid piece of matter (usually ice) restricting the flow of liquid nitrogen.

Solution
Take the leg out of the Dewar vessel. Wait 15 minutes for the end of the leg to warm up to room temperature. Disconnect the dry air shroud gas from its connector at the top of the nozzle and reconnect it to the SUCT connector on top of the Coldhead. Set the gas flow to about 5 L/min. This will blow air down the Cryostream transfer line and will both remove any solid matter or ice from the line and dry it out. This should be done for about half an hour until the leg has warmed up. Once the block is cleared reconnect the tubing to its correct connectors.

9.2.12 Gas Pump Unit is making a strange noise

Cause
Although sounds are often difficult to interpret it is worth noting a few things. The noises from the Gas Pump Unit vary from pump to pump and voltage to voltage. If you are unhappy with the noise your pump is making, possible causes are:

1. A rattling or knocking can often indicate a broken con rod.

2. A grinding usually indicates a failed bearing.

3. A slapping noise can be produced by the diaphragm. **IT IS NOT USUALLY A FAULT**.

Solution
Contact your local agent or Oxford Cryosystems to get the replacement part or the pump serviced.

9.2.13 Flow rate will not rise to 10 l/min when the TURBO button is pressed

Symptoms
The system is running and when the TURBO button is pressed, the flow does not rise from 5 L/min to 10 L/min. The flow may stop at 7 or 8 L/min.

Cause
One of the most likely causes of this problem is that the unit in the Controller regulating and monitoring the flow is not functioning correctly. To test for this, do the following:

1. Disconnect the SUCT and FLOW tubes from their connectors on the top of the Coldhead. (You will need to remove the tube from its compression fitting too.)

2. Switch the Controller on, (if it is not on already!)

3. Program in a RAMP to whatever the current room temperature default reading is (about 290 K to 295 K) and press START.

4. Once the system has been running for a minute or so, press the TURBO button several times to cycle the flow rate between 5 and 10 L/min.

5. Press STOP when you have finished.

If the flow does not rise to 10 L/min then there is likely to be a problem with the Flow Controller.

Solution
Contact Oxford Cryosystems for a replacement Flow Controller.

9.2.14 EPROM fail on initialisation

Symptoms
During the initialisation of the Controller, there is an EPROM failure.

Cause
The likely cause of this problem is corruption of the EPROM software. If this is the case, this problem can be rectified with the assistance of Oxford Cryosystems.

Take the lid off the Controller, be careful of the earth wire that is attached inside when doing so. Move the jumper (JP9) to the SET pins and then turn the Controller on.

Once the Controller has finished initialising, you will see a list of parameters like those listed below. Write down all the parameters and their corresponding values and send them to support@oxcryo.com.

Firmware	Date
Board	Coldhead
Fan	Control
Suct H	SuctSet
Evap H	TestR
Gas Heat	SC.SUCT
Gas Flow	RC.SUCT
Shutdown	SC.EVAP
T Units	RC.EVAP
Cool T	SC.GAS
Hours	RC.GAS

Solution

Oxford Cryosystems will then contact you letting you know if any of these numbers are wrong the how to edit them.

9.2.15 Flashlight test

To be sure the flow rate of the outer dry air stream is correct, it is often better to set the flow by eye rather that by trying the guess what the flow should be by looking at the numbers.

Turn all the lights off in the x-ray room and shine a flashlight up towards the nozzle of the Cryostream in an attempt to highlight the plume created by the cold gas stream. As the gas stream leaves the nozzle it is really made up of two parts; the first 'invisible' 10 or 12 mm and the remaining plume of ice. The object of the exercise is to maximise the length of the 'invisible' section. This should only be done over the first 15 L/min of air from the dry air source. One should not be fooled into thinking that at 25 L/min there is no plume, and therefore, no ice because the ice will build rapidly around the end of the nozzle and blow the sample from its support.

10 Technical Support

To allow Oxford Cryosystems to offer fast and accurate technical support, please quote your Cryostream Serial Number with all technical issues. It is worth keeping a record of this number in a convenient place:

CRYOSTREAM SERIAL NUMBER
This Cryostream serial number is ________

Before you return your equipment you must warn Oxford Cryosystems by contacting us.

Oxford Cryosystems Ltd contact details:

Email: support@oxcryo.com

Phone: +44 (0)1993 883488

Fax: +44 (0)1993 883988

10.1 Returns procedure

Use the following procedure to return ANY items for repair.

1. Contact Oxford Cryosystems and obtain an 'RMA' number for your equipment which must be written on each box that you return. Without this number we may reject packages. You will also be emailed a form which you must fill in and email or fax back to us prior to sending your package(s).

2. Remove all traces of dangerous substances and any accessories that will be returned to Oxford Cryosystems. Drain all fluids and lubricants from the equipment and its accessories.

3. Disconnect all accessories from the equipment. Safely dispose of the filter elements from any oil mist filters.

4. Seal up all of the equipment's inlets and outlets (including those where accessories were attached). You may seal the inlets and outlets with blanking flanges or heavy gauge PVC tape.

5. Seal contaminated equipment in a thick polythene bag. If you do not have a polythene bag large enough to contain the equipment, you can use a thick polythene sheet.

6. If the equipment is large, strap the equipment and its accessories to a wooden pallet. Preferably, the pallet should be no larger than 510 mm x 915 mm (20"x 35"); contact Oxford Cryosystems if you cannot meet this requirement.

7. If the equipment is too small to be strapped to a pallet, pack it in a suitable strong box.

8. If the equipment is contaminated, label the pallet (or box) in accordance with laws covering the transport of dangerous substances.

Oxford Cryosystems - Warranty Certificate

This warranty is subject to the Oxford Cryosystems Ltd's (OCL) Terms and Conditions of Sale.

OCL warrants to the Buyer that the goods sold for use hereunder will be free from defects in material and workmanship under normal use and operation for 12 months from the date of shipment from OCL's premises.

In order to obtain the benefits of the warranty the Buyer must first notify OCL of the defects. An OCL representative will verify the nature of the defect and if it is covered by this warranty, OCL will issue the Buyer with a RMA number and provide the Buyer with instructions on how to return the goods to OCL. The Buyer must return the goods according to instructions from OCL, complete with a written description of the claimed defect and RMA number. The goods should be packed safely, preferably in its original packaging prior to return.

The Buyer shall meet the cost of shipping the defective goods to OCL and OCL will pay any return costs to the Buyer

OCL's obligation under this warranty is limited to its option to repair or replace goods that are proven to be defective when used under normal operating conditions and within specification.. This warranty does not cover any changes made by the customer, depreciation of the goods or claims for compensation.

No warranty is given for damage resulting from misuse or fair wear and tear. In addition, this warranty does not cover any costs incurred in damage arising from the dismantling or reassembly of any of the goods, or for consequential losses of time or materials caused by Cryostream failure.

Registration

In order for us to be able to provide fast and effective service, you should register your system with us. Please send the serial number of the system (found engraved on the Coldhead) to support@oxcryo.com, together with your full contact details.

To make contact with Oxford Cryosystems you can telephone, fax, or email us at:

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Lower Road, Long Hanborough
Oxford OX29 8LN, UK
Tel: +44 1993 883488 Fax: +44 1993 883988
Email: info@oxcryo.com